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Indian Standard

METHODS OF TESTING PLASTICS

PART I CHARACTERIZATION OF POLYMER STRUCTURE AND SIZE

Section 1 Determination of Molecular Mass from Viscosity

UDC 678·7 : 541·28·08 : 532·13

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METHODS OF TESTING PLASTICS

PART I CHARACTERIZATION OF POLYMER STRUCTURE AND SIZE

Section 1 Determination of Molecular Mass from Viscosity

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Indian Standard

METHODS OF TESTING PLASTICS

PART I CHARACTERIZATION OF POLYMER STRUCTURE AND SIZE

Section 1 Determination of Molecular Mass from Viscosity

0. FOREWORD

0.1 This Indian Standard (Part I/Sec 1) was adopted by the Indian Standards Institution on 25 October 1978, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Methods of determination of molecular mass may be divided into two classifications, namely, primary and secondary. The former includes osmotic pressure and other colligative techniques, electronmicroscopy, gel permeation chromatography, ultracentrifugal and end-group analysis. In the secondary methods solution viscosity is widely used because of the simplicity of technique and apparatus. It is also used as a process control method. It is applicable to all polymers that dissolve completely without chemical reaction or degradation to form solutions that are stable at temperature between room temperature and about 150°C.

0.3 The determination of dilute-solution viscosity provides one item of information towards the molecular characterization of polymers. When viscosity data are used in conjunction with other molecular parameters, the properties of polymers depending on their molecular structure may be better predicted.

0.4 With certain restrictions, satisfactory correlations can be obtained between dilute-solution viscosity and molecular parameters, such as molecular mass or chain length. The most limiting restrictions which should be observed are as follows.

0.4.1 It must be known that the polymers used to establish the correlations and those to which they are applied do not consist of or contain branched species. Basically a measure of molecular size and not molecular mass, the dilute-solution viscosity may be correlated appropriately with the molecular mass or chain length only if there is a unique relationship between the mass and the size of the dissolved polymer molecules. This is the case for linear, but not for most branched polymers. Caution should be exercised in applying empirical viscosity-molecular mass relationships to polymers of unknown or uncertain structure since unsuspected branching may exist.

0.4.2 For reasons similar to those outlined in **0.4.1**, it must be required that the polymers to which the correlations are applied have the same chemical composition as those used in establishing the relationships.

0.5 For polymers meeting the restrictions of **0.4**, empirical relationships can be developed between the dilute-solution viscosity of a polymer and its hydrodynamic volume or average chain dimension (radius of gyration or end-to-end distance). Such relationships depend upon any variables influencing the molecular size of the dissolved polymer. The most important of these variables are solvent type and temperature. Thus, the solution viscosity of a given polymer specimen depends on the choice of these variables, and they must always be specified with the viscosity for complete identification.

0.6 The further empirical interpretation of viscosity data in terms of molecular mass rather than size requires control of the variables described in **0.4.**

0.7 The solution viscosity of a polymer of sufficiently high molecular mass may depend on rate of shear in the viscometer, and the viscosity of a polyelectrolyte (polymer containing ionizable chemical groupings) will depend on the composition and ionic strength of the solvent. Special precautions beyond the scope of this method are required when measuring such polymers.

0.8 Finally, the viscosity of polymer solutions may be affected drastically by the presence of recognized or unrecognized additives in the sample, including but not limited to colourants, fillers, or low-molecular mass species.

0.9 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I/Sec 1) prescribes method for determination of the dilute-solution viscosity of polymers and the calculation of molecular mass.

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*Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 2828-1964* shall apply.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals which do not contain impurities that affect the results of analysis.

4. APPARATUS

4.1 Volumetric Flasks — 100-ml or other size found convenient.

4.2 Transfer Pipettes — sizes between 1 and 25 ml, as required. Transfer pipettes for use with polymer solutions should have about 2 mm cut from their lower tips to permit more rapid transfer of the solution to the viscometer.

4.3 Constant Temperature Bath — capable of maintaining ± 0·1°C at the desired operating temperature (usually between 25 and 135°C). Less stringent temperature control (± 0·2°C) is satisfactory upon demonstration that the precision of the results is not affected.

4.4 Viscometer — glass, capillary type. Efflux time for the solvent and temperature used shall be greater than 200 seconds (except that efflux time for semi-micro viscometers shall be greater than 80 seconds), to eliminate the need for kinetic-energy corrections.

NOTE — Two types of viscometers, commonly used are as follows:

The first type is a constant-volume device of simple construction, recommended for use where solution viscosity is to be measured at a single concentration, as for determination of the viscosity number or logarithmic viscosity number. It may also serve for the determination of the limiting viscosity number through measurement of several solutions having different concentrations.

The second type of viscometer, commonly called a dilution viscometer, is a time-saving device for the determination of limiting viscosity number since it does not require constant liquid volume for operation. Several concentrations of a polymer solution can be tested by adding a known quantity of the solvent at the test temperature directly to the viscometer, mixing, measuring the viscosity and then making the next dilution. The viscosity of the pure solvent shall be measured separately.

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*Glossary of terms relating to plastics industry.

†Specification for water for general laboratory use (*second revision*).

An alternative procedure is to start with the minimum volume of the pure solvent, then add aliquots of a concentrated stock solution to the viscometer to obtain values of the viscosity ratio at successively higher concentrations. The choice of procedures is dictated by the range of volumes with which the viscometer operates and the range of concentrations desired for test.

4.5 Timer — graduated in divisions of 0·1 second or less.

4.6 Thermometer — suitable for the specified test temperature and conforming to IS : 4825-1968*.

4.7 Fritted Glass Filter Funnel — coarse grade.

5. MATERIAL

5.1 Solvents as required or as recommended in Appendix A.

5.2 Heat-Transfer Liquid — for constant-temperature bath.

NOTE — The following materials have been found suitable as heat transfer liquids:

a) Silicone oil;
b) Mineral oil;
c) Groundnut oil;
d) Water; and
e) Water-miscible liquid, such as glycerol or ethylene glycol.

The material selected shall not discolour or smoke on prolonged selected exposure at the test temperature; in some cases discolouring may be inhibited by the use of an antioxidant. The use of water or water-miscible liquid facilitates cleaning glassware used in the test.

5.3 Nitrogen — for purging.

6. PREPARATION OF SAMPLE

6.1 The sample need not be predried or conditioned unless previous specific knowledge, for example that the material is known to be hygroscopic, makes such pretreatment desirable (*see* Appendix A).

6.2 If it is known that the sample dissolves only slowly in the selected solvent, pretreatment to reduce the particle size of the sample may be advisable.

NOTE — Some samples can be pulverized conveniently in a rotary cutting mill with a 20-mesh screen at the outlet of its pulverizing chamber. The Wiley mill, widely available from scientific supply houses, has been found satisfactory. Care should be taken to avoid overheating the sample during pulverization, which might lead to thermal degradation. Low-melting polymers, or hard, tough samples, often may be satisfactorily pulverized only at very low temperature, for example, in the presence of dry ice or liquid nitrogen (*see* Appendix A).

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*Specification for laboratory and reference thermometers.

7. PROCEDURE

7.1 Weigh the sample (*see* Appendix A for size and tolerance) in a tared 100-ml volumetric flask (or weigh and transfer quantitatively to the flask). If the sample is known to oxidize easily in the subsequent dissolving step, the flask may be purged with nitrogen.

NOTE 1 — Other sizes of volumetric flasks may be used, depending on viscometer size and amount of sample available. If another size flask is used, adjust the sample weights, solvent and solution volumes accordingly.

NOTE 2 — For greatest reliability of results and in the absence of specific information (*see* Appendix A), select the sample size on the basis of experience to give a viscosity ratio near 1·5. If several concentrations of a solution of a single sample are to be used (*see* Note 3), select them so that the viscosity ratio falls in the range from 1·2 to 2·0.

NOTE 3 — Preparation of a single solution may often suffice, either for determining the viscosity ratio or logarithmic viscosity number, or as a stock solution for use in a dilution viscometer to determine the limiting viscosity number. If more than one solution concentration is desired, weigh a series of specimens (often four) into separate flask, selecting the specimen weights to give the desired solution concentration. Duplicate specimens may be prepared if desired.

7.2 Add approximately 50 ml of solvent to each solution flask, purge with nitrogen if necessary, and shake the flask once every 10 minutes until solution is complete (alternatively, use a laboratory shaker). Elevated temperature may enhance solution rate (*see* Appendix A) but should be used with caution since some polymers and solvents have limited high-temperature stability. If solution is carried out at an elevated temperature subject the flask of pure solvent to the same conditions as the polymer solution.

NOTE — Complete solution of all the specimens is essential. When solution appears complete, examine the flask with care to be sure that no undissolved material, gel particles, or foreign matter is present.

7.3 Place the volumetric flask containing the solution and the pure solvent in the constant-temperature bath maintained at the test temperature. Add solvent to the solution flask to a total volume of about 99 ml using solvent maintained at the bath temperature. Mix the solution thoroughly, and replace in the bath. After temperature equilibrium has been achieved (10 to 30 minutes) complete the dilution to the 100-ml mark by adding solvent maintained at the test temperature, using a transfer pipette. Mix the contents of the flask thoroughly.

NOTE — Be sure that the solution is uniformly mixed. If the test temperature is above ambient, avoid cooling the flask excessively while mixing.

7.4 Where necessary to prevent oxidation, purge the viscometer with a slow stream of nitrogen. With the viscometer permanently positioned in the constant-temperature bath at the required temperature, transfer a suitable amount of solution into the viscometer using a suitably modified transfer pipette. The amount varies with the type of viscometer used. If necessary, filter the solution through a fritted-glass filter or equivalent into the viscometer taking care not to lose solvent in the process (pressure filtration). Filtration is usually desirable.

NOTE — If the solution is to be handled at elevated temperatures, the transfer pipette may be fitted with a suitable heating mantle to retard precipitation of polymer from the solution during the transfer.

7.5 After temperature equilibrium has been achieved (a minimum of 10 minutes), bring the liquid level in the viscometer above the upper graduation mark by means of gentle air (or preferably, nitrogen) pressure applied to the arm opposite to the capillary. Allow the solution to drain through the capillary. Start the timer exactly as the meniscus passes the upper graduation mark and stop it exactly as the meniscus passes the lower mark.

7.6 Determine the efflux time at least three times each for the solution and for the pure solvent. Three consecutive readings should agree to within 0·1 second or 0·1 percent of their mean, whichever is greater. Larger variations may result from foreign material in the viscometer or from inadequate temperature control and require repetition of the experiment after their cause is located and corrected.

8. CALCULATIONS

8.1 Viscosity Ratio — Calculate the viscosity ratio from the average efflux time for the solvent, t_0 and the average efflux time for the solution t, as follows:

$$\text{Viscosity ratio} = t/t_0$$

NOTE — Strictly, the viscosity ratio is defined as η/η_0 where η, η_0 are the viscosities of the solution and solvent respectively, and are related to the corresponding efflux time by:

$$\eta = ct\sigma - E\sigma/t^2$$
$$\eta_0 = Ct_0\sigma_0 - E\sigma_0/t_0^2$$

where C and E are constants for the particular viscometer used. The equation in **8.1** follows if the second term in these relations, a kinetic-energy correction, is negligible, and the respective solvent and solution densities, σ_0 and σ are substantially equal. The kinetic-energy correction is negligible for the recommended viscometers and efflux times.

8.2 Logarithmic Viscosity Number — Calculate the logarithmic viscosity ratio for each solution concentration as follows:

$$\text{Logarithmic Viscosity Number} = l_n\ (\eta/\eta_0)/C$$

where l_n (η/η_0 is the natural logarithm of the viscosity ratio and C is the solution concentration in grams per millilitre (grams per millilitre of solution). The units of logarithmic viscosity number are, therefore, ml/g.

8.3 Limiting Viscosity Number — Plot the logarithmic viscosity number versus concentration, for several solution concentrations, on rectilinear graph paper. Draw the best straight line through the points and extrapolate it to zero concentration. The limiting viscosity number (η) is the intercept of the line at zero concentration.

NOTE 1 — The viscosity number $(\eta - \eta_0)/\eta_0 C$ may be calculated and plotted versus concentration on the same graph with the logarithmic viscosity number. The two lines should extrapolate to the same point (the limiting viscosity number) at $C = 0$; plotting both functions may serve to fix the limiting viscosity number with greater accuracy. If the limitation on viscosity ratio stated in Note 2 (**7.1**) is observed, the extrapolation lines should be accurately straight.

NOTE 2 — For some polymer-solvent systems, it may be demonstrated that the slopes of the lines of viscosity number and logarithmic viscosity number versus concentration are closely similar for all samples normally encountered.

9. PRECISION AND ACCURACY

9.1 For most polymer-solvent systems, the logarithmic and limiting viscosity numbers may be determined with a standard deviation for reproducibility of about 1·0 ml/g. Since the viscosity numbers are available only from these measurements, accuracy of their values has no meaning.

10. REPORT

10.1 The report shall include the following.

10.1.1 Complete identification of the sample tested.

10.1.2 Conditioning procedure, if any.

10.1.3 One or more of the following:

a) The viscosity ratio, given to one significant figure beyond the decimal point followed by the concentration of the test solution in g/ml.

b) The logarithmic viscosity number in ml/g carried to one significant figure beyond the decimal point followed by the concentration of the test solution in g/ml.

c) The limiting viscosity number in ml/g, carried to the decimal point.

10.1.4 The solvent employed and the test temperature.

NOTE — ISO Draft Recommendation No. 1628 Directives for the standardization of methods for the determination of the dilute solution viscosity of polymers, differs from this method in the following respects:

a) Application of kinetic-energy corrections are different such that errors in viscosity ratios may be as high as 3 percent depending on flow times and viscometer type, and

b) The recommended test temperature is 27 ± 0·1°C.

It is, therefore, recommended that caution be exercised when comparing data obtained by both methods.

APPENDIX A

(*Clauses* 5.1 and 6)

DETAILS OF MATERIALS AND PROCEDURES FOR IMPORTANT POLYMER TYPES

A-1. POLYAMIDE

A-1.1 Recommended solvents are formic acid (90 ± 0·2 percent in water) or *m*-cresol, melting point 11 to 12°C.

A-1.2 Recommended dissolving conditions are for formic acid, 30°C; for *m*-cresol, 2 hours *Max* at 95 to 100°C or 8 hours at 50°C.

A-1.3 The recommended test temperature is 27 ± 0·1°C.

A-1.4 The recommended solution concentration is 0·005 0 ± 0·000 02 g/ml.

A-2. POLYCARBONATE

A-2.1 Recommended solvents are methylene chloride (purified grade) and dry *p*-dioxane, 60°C.

A-2.2 Recommended dissolving temperatures are; for methylene chloride, 30°C; for *p*-dioxane, 60°C.

A-2.3 If pigments or other fillers are present, the resin shall be dissolved in methylene chloride and the solution filtered. The methylene chloride shall be evaporated from the resulting clear solution to leave the resin in the form of a thin film. After this has been dried for several hours at 125°C, samples shall be taken for viscosity measurement.

A-2.4 The recommended test temperature is 27 ± 0·1°C.

A-2.5 The recommended solution concentration is 0·004 0 ± 0·000 2 g/ml, or by convenient dilution from 0·010 ± 0·000 02 g/ml.

A-3. POLY (ETHYLENE) TEREPHTHALATE

A-3.1 Recommended solvents are *o*-chlorophenol (melting point 0°C) or a mixture of 1 part trichloroacetic acid and 3 parts dichloromethane. Solvents should contain less than 0·15 percent water.

CAUTION — Since *p*-chlorophenol is toxic, appropriate measures must be taken to avoid breathing its vapours or contact of the liquid with skin or eyes.

A-3.2 The samples should be dried to a water content of 0·03 percent or less. Drying may be carried out at 100°C for about 3 hours under a pressure of 1 mmHg or less.

A-3.3 Recommended dissolving conditions are 2 to 3 hours at 90 to 100°C.

A-3.4 The recommended test temperature is 27 ± 0·1°C.

A-3.5 The recommended solution concentration is 0·005 0 ± 0·000 2 g/ml.

A-4. POLY METHYLMETHACRYLATE

A-4.1 Recommended solvent is 1, 2-dichloroethane (ethylene dichloride), reagent grade.

A-4.2 Pretreatment of the sample should not include cutting or grinding in such a way as to cause shear degradation of the polymer.

A-4.3 Recommended dissolving conditions are 24 hours at 30°C.

A-4.4 The recommended solution concentration is 0·002 0 ± 0·000 02 g/ml; but the recommendations of Note under **7.2** should be construed to supersede this.

NOTE — The limiting viscosity number may be estimated from data obtained at a single concentration by use of the following equation:

$$(\eta) = (2^{1/2}/C)\left[\eta/\eta_0 - 1 - l_n(\eta/\eta_0)\right]^{1/2}$$ (*see* **8.1** and **8.2** for explanation)

A-5. POLY (VINYLCHLORIDE)

A-5.1 The recommended solvent is cyclohexanone, boiling point 155 to 156°C (760 mmHg).

A-5.2 Samples should be dried to a moisture content below 0·1 percent.

A-5.3 The recommended dissolving temperature is 85 ± 10°C.

A-5.4 The recommended test temperature is 27 ± 0·1°C.

A-5.5 The recommended solution concentration is 0·002 0 ± 0·000 02 g/ml.

NOTE — The limiting viscosity number may be estimated from data obtained at a single concentration by use of the following equation:

$$(\eta) = \tfrac{1}{4}\left[(\eta - \eta_0)/\eta_0 C\right] + \tfrac{3}{4}\left[l_n(\eta/\eta_0)/C\right]$$ (*see* **8.1** and **8.2** for explanation)

A-5.6 Precision

A-5.6.1 *Repeatability* — Duplicate results by the same analyst should not be considered suspect unless they differ by more than 1 percent.

A-5.6.2 *Reproducibility* — The average result of two determinations reported by one laboratory should not be considered suspect unless it differs from that of another laboratory by more than 2 percent.

(*Continued from page* 2)

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8543 (Part II/Sec 2)-1977	Methods of testing plastics : Part II Testing of materials before moulding, Section 2 Determination of apparent density of moulding material that cannot be poured from a funnel
8543 (Part II/Sec 3)-1977	Methods of testing plastics : Part II Testing of materials before moulding, Section 3 Determination of bulk factor of moulding material
8543 (Part XIII/Sec 1)-1977	Methods of testing plastics : Part XIII Tests for specific products, Section 1 Methods for sampling and tests for plastics buttons (thermosetting) (superseding IS : 1465)